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CHARACTERIZATION OF BERYLLIUM

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CHACTERIZATION

OF

BERYLLIUM

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Division of Engineering - National Research Council

as a service of

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The National Academy of Engineering

to the

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National Research Council

National Academy of Sciences - National Academy of Engineering

Washington, D. C.

August 1966

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ABSTRACT

Research and development projects involving beryllium have not always controlled or recorded all the significant variables. Applications have languished because of lack of reproducibility or the difficulty of relating unexpected behavior to prior processing. What has been lacking is an adequate characterizing of the metal. While this attempt at characterizing has fallen short of a complete scientific description, we have reached the stage where major correlations between processing, structure, and properties can be obtained if the data indicated in the report is collected.

This brief report has compiled the major process variables corresponding to the principal operations gone through in producing a beryllium mill product, and then a part. This compilation comprises a check list which should be reviewed at the start of a research or development effort to ensure that variables which might be significant are not being overlooked. The ultimate object of compiling and studying such data is to assure uniformity, and thus introduce reliability into manufactured parts.

CONTENTS

CHARACTERIZATION OF BERYLLIUM

In a review of beryllium research*, the statement was made, "It is the unanimous opinion of the Committee that future research support should place emphasis on the characterization of polycrystalline material.". The report went on to state, "Characterization is viewed broadly as a correlation of processing factors (such as casting, attritioning, and consolidation variables) and microstructure, (the latter being defined in terms of light and electron microscopy, microprobe analysis and texture determined by X-ray analysis) and a correlation between microstructural changes and those engineering properties that are used to predict performance.".

A task group was established by the Committee on Beryllium Metallurgy in February 1966 to attempt to determine if the characterization of beryllium was possible, and if so, to suggest how this might be done. The members of this task group are shown on Page v of this report. Substantial agreement was obtained, as reported in the following pages.

It was considered that a full scientific characterization would be both extremely difficult and of little practical utility. It should be noted that this is, instead, an attempt to reconcile the most serious problems of beryllium with a practical approach to a better understanding of the relationship between process, structure, and properties. As such, this brief report represents an engineering approach (as contrasted to a purely scientific approach). It is strongly felt that improved intercourse among the users, fabricators, and producers of beryllium (under the guidance of the various sponsoring agencies) can and will result in substantially improved usage of this potentially valuable metal.

An MAB Ad Hoc Committee on Characterization of Materials has proposed the following definition:

> Characterization describes those features of the composition and structure of a material that are significant for a particular preparation, study of properties, or use, and suffice for reproduction of the material.

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*Fifth Progress Report by the Committee on Beryllium Metallurgy of the Materials Advisory Board (MAB-199-M(5)), February 1966.

This report does not purport to characterize beryllium; rather it attempts to provide guidance to research directors and project sponsors, describing the measurements which should be made so that a true characterization can be performed later. The emphasis here is on those processing steps which probably have the greatest influence on structure, and thus on properties.

The approach used was to visualize the processing and fabrication of beryllium as a framework, beginning with the reduction to metal through to the manufacture of a part or component, as shown in Figure 1. The principal steps involved are shown on the chart, Table I. At each major operation, called a checkpoint, two classes of descriptors are listed. Under Roman numerals are the measurements which describe the structure or properties. Under Arabic numerals, the process variables which are measured or controlled are listed. In each class, typical measurements or variables which are believed to be important, are listed. This listing is not all-inclusive but is suggestive of major variables. The tabulation can be considered as a checklist of items to be reviewed when planning a research program. It is unlikely that funds would be available to measure or describe all the items listed, nor that all would be pertinent for any specific program.

In compiling these lists, consideration was given to the problem of specifying both processing and minimum level of properties when procuring materials. The point to be emphasized is that, at the least, as many as possible of these important variables should be measured and recorded (even if not controlled) to permit possible later correlation with structure and performance. The purchaser may wish to specify the processing, but may not then also expect a guaranteed level of properties.

As an example of how this scheme might work, let us suppose that an experimental program is to be planned with the goal of developing jet engine hardware.

Two specific vacuum hot pressed billets (two compositions) would be prepared (special material, special handling). The material from which they are made should be fully characterized, i.e., all process parameters

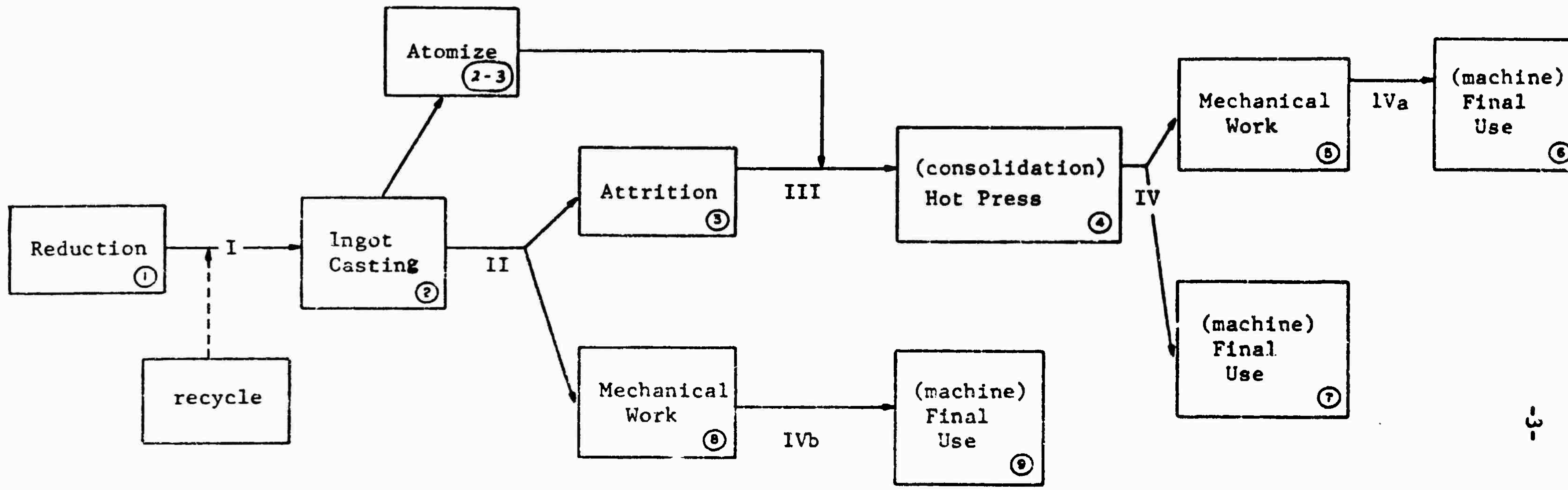

Figure 1. Flow chart for beryllium production. See Table I for description of process variables (Arabic numerals) and checkpoints for measurements (Roman numerals).

Table I

Processing Steps & Associated Measurements

CHECKPOINT; MEASUREMENTS OF STRUCTURE AND/OR PROPERTIES	CORRESPONDING PROCESS VARIABLES
I. After Reduction, Before Ingot Casting Chemical Composition	1. Reduction
	2. Ingot Casting Crucible charge (virgin flake or pebble, remelt or revert scrap) Crucible composition and preparation Atmosphere (time, temperature, vacuum) Pour temperature and method Mold (material, preheat, configuration, hot-top) Solidification rate
	2-3. Atomization Crucible material Atmosphere Temperature Atomization medium Collection medium
II. After Ingot Casting, Before Attritioning or Hot Working Chemical composition (average, local) ——— The following checkpoints apply to metal to be worked: Soundness (cracks, inclusions, voids) Morphology (size, shape distribution) Macrostructure (etched slice) Density Inclusions and precipitates (fractography) Texture	3. Comminution Atmosphere Grinding medium Mill materials Classification method (wet, dry, etc.) Temperature System to ensure detection of extraneous particles

III. <u>After Comminution, Before Hot Pressing</u>

- Particle size, shape, distribution
- Chemical Composition
- Extraction of undesirables (liberation of second phases)
- Shelf life (date of manufacture)
- Tramp inclusions

4. <u>Hot Pressing</u>

- Cycle parameters (time, temperature, atmosphere)
- Container and die materials and preparation
- Loading method (preconsolidation, vibration, etc.)
- Pressing size
- Sectioning plan
- Date of pressing
- Amount of skin removed
- Can material and billet density (for direct consolidation of powder)
- Reduction per pass
- Direction of working
- Deformation rate
- Cooling rate
- System for foreign particle avoidance

IV, IVa, IVb. <u>After Hot Pressing, After Mechanical Working</u>

- Chemical composition (average, local)
- Soundness (cracks, inclusions, voids)
- Morphology (size, shape, distribution)
- Macrostructure (etched slice)
- Density
- Inclusions and precipitates (fractography)
- Texture
- Mechanical properties
- Physical properties (resistivity, conductivity, elastic constants)

5,8. <u>Mechanical Work</u>

- Time
- Temperature
- Percent reduction
- Working directions and sequence
- Intermediate stress relief or anneal
- Atmosphere
- Container materials
- Lubrication
- Protection from contamination
- Die design
- Restraint or methods of hydrodynamic compression

6,7,9. <u>Final Use</u>

- Failure analysis of overstressed part
- Fractography
- Metallography (grain size and shape, inclusions, microstructural constituents)
- Specific inclusions (size, shape, distribution, and composition)
- Radiography
- Ultrasonic examination

should be carefully documented and the material should have been known to possess adequate forgeability. For each of the two billets, two working processes would be utilized which are designed to give different degrees and types of texturing. The two working processes would be established to give extremes in texturing. For each of the two working processes, for each of the two billets, the following mechanical tests would be run and data gathered for the jet engine designer: tensile test results, low cycle fatigue, high frequency fatigue, impact, strain rate sensitivity, creep strength. The preceding properties should be obtained in both smooth and notched test bars. Fractography would be employed to establish the precipitates that are formed as a result of extremes of heat-treatment. The test forgings should be sufficiently large in size that post-forging heat treatments can be tried on a second series of test bars.

In contrast, a sheet rolling study might utilize a single starting material, particularly if funds are quite limited. Similarly, a single casting, comminution, and pressing practice might be adopted (with all pertinent measurements of these variables recorded and reported). The variables might principally be those of mechanical work, where the factors of temperature, reduction per pass, cross-rolling, annealing, etc. would be studied. After trials involving some of the variables shown in the Table as 5,8, a selection of the measurements (composition, soundness, etc.) shown as IV, IVa, IVb would be made and reported. If a good selection of the processing variables and of the structure and properties has been made, a step forward in the characterizing of beryllium will have been accomplished.

The use of standardized test procedures is highly recommended. Attention is called to the report of the Subcommittee on Test Methods of the Committee on Beryllium Metallurgy (MAB-205-M, "Evaluation Test Methods for Beryllium"). Only through the use of consistent methods can statistical data be generated from which correlations of processes, structures, and properties be obtained.

Even a cursory review of the numerous variables involved in the preparation of mill forms and the conversion of such forms to hardware

suggests the advisability of conducting research with metal especially prepared. Commercially available metal would inherently involve variability which might obscure the desired results from the experiments. In all probability, complete and accurate processing records are not made, nor would commercial policy permit them to be provided when buying a commercial product. For special research programs, it should be possible to specifically indicate the input material and the processing steps which are required to convert the raw material into a polycrystalline form suitable for subsequent fabrication and testing.

Existing knowledge does not permit one to list those processing variables which have the greatest influence on structure and those structural variations which have the greatest influence on mechanical properties. The items listed in this attempt to characterize probably include most such items. Only through research programs in which this kind of data is obtained will it be possible to establish a much smaller list of the truly significant variables. More uniform material with greater reproducibility could be the outcome. This could lead to the development of material.

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